REVISED SYNOPSIS

SYNTHESIS, SPECTROSCOPIC, KINETIC AND BIOLOGICAL STUDIES OF COPPER (II) SESAME & COPPER (II) GROUNDNUT SOAPS AND THEIR COMPLEXES

ताम्र (II) तिल एवम् ताम्र (II) मूँगफली साबुनों व उनके संकुलो का संश्लेषण, स्पेक्ट्रोमितीय, गतिकीय और जैविक अध्ययन

A

SYNOPSIS

SUBMITTED TO

M.D.S. UNIVERSITY AJMER

FOR DOCTOR OF PHILOSOPHY

IN FACULTY OF SCIENCE

(CHEMISTRY)

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Submitted by :

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FORMAT FOR OUTLINE (REVISED SYNOPSIS) OF PROPOSED RESEARCH WORK

(To be supplied in one copy)

1.	Name of scholar	(In English)	:	ASHA MEENA
		(In Hindi)	:	आशा मीणा
2.	Title of the Research Work			
		(In English)	:	SYNTHESIS, SPECTROSCOPIC, KINETIC AND BIOLOGICAL STUDIES OF COPPER (II) SESAME & COPPER (II) GROUNDNUT SOAPS AND THEIR COMPLEXES
		(In Hindi)	:	ताम्र (II) तिल एवम् ताम्र (II) मूँगफली साबुनों व उनके संकुलो का संश्लेषण, स्पेक्ट्रोमितीय, गतिकीय और जैविक अध्ययन
3.	Location			
	a. Institution/Department where the work is to be done.		:	S.P.C. GOVT. COLLEGE, AJMER
	b. Geographical area of investigation		:	M.D.S. University, Ajmer University of Rajasthan , Jaipur S.P.C. Govt. College, Ajmer S.D. Govt. College, Beawar.
4.	Introduction Importance of proposed Investigation, objectives.		:	ENCLOSURE - 1
5.	Review (work already done on the Subject of proposed research study).		:	ENCLOSURE - 2
6.	Research gaps identified in the Proposed field of investigation (Based on the review)		:	ENCLOSURE - 3
7.	Major hypothesis, if any		:	ENCLOSURE - 4
8.	Research methodology		:	ENCLOSURE - 5
9.	List of references		:	ENCLOSURE - 6

7/9/16

Signature of the candidate with date
(ASHA MEENA)

7/9/16

Outline of the synopsis approved.

Lecturer

Name and signature of supervisor with date and seal
(Dr. Rashmi Sharma)

Govt. College, Ajmer

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REVISED SYNOPSIS

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Asha Meena

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ENCLOSURE-1

INTRODUCTION IMPORTANCE OF PROPOSED INVESTIGATION, OBJECTIVE

Oils consist of wide group compounds that are soluble in organic solvents and insoluble in water. They have low density than water at normal room temperature range in consistency from solid to liquid, depending on their structure and composition[1]. Various edible oils like Groundnut, Sesame and non-edible oils like Neem, Karanj are widely used for medicinal purpose[2].

The Copper (II) Soaps in polar and non-polar solvents having remarkable interest and find their uses in various fields of applications like foaming, wetting, emulsification and lubrication etc due to the surface active properties of soaps[3,4]. Copper Soaps and their complexes have many applications in different fields like wood preservation, dyeing, detergency, rot-proofing, textiles, paints, electroplating, wetting, foaming, herbicidal, emulsification and agrochemicals[5-14].

Copper Soaps have a tendency of complexation with nitrogen and sulphur containing ligands. Synthesized Copper Soaps and their complexes may play a significant role in biological activities and have sufficient pharmaceutical, industrial and analytical applications[15].

Thermogravimetric methods have been used in characterization of oils and fats. This technique has been employed to study the kinetics by thermal decomposition of soaps and complexes derived from these oils[16-18].

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In the proposed study the synthesis of Copper Soaps derived from edible oils will be done to understand structural aspects, kinetic parameters through spectrophotometric or thermal methods, reactivity order, activation energy of their soaps and complexes with ligands. Their biological studies will also give an important information about their industrial utilization.

The main objectives of the proposed research work are as follows :-

1. Synthesis of Copper Soap derived from various edible oils like Sesame, Groundnut.
2. Purification of Soaps.
3. Synthesis of ligand.
4. Purification of ligand.
5. Synthesis of complexes.
6. Purification of complexes.
7. Spectroscopic studies like IR, NMR and ESR of Copper complexes.
8. Thermogravimetric analysis and Photocatalytic Degradation of synthesized Copper complexes.
9. Biological studies of Copper complexes.

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Chemical and thermal stability of rice husks against alkali treatment have been done[27]. Biological activities of some new environmentally safe 2-aminobenzothiazole complexes of Copper (II) derived under microwave irradiation has been studied earlier[28]. Interpretation of relationship between density and micellization of Copper (II) Soap complexes in binary solvent mixture has been already done[29].

Synthesis and characterization of Copper metal soaps from *Thevetia peruviana* and *Hura crepitans* seed oils has been done[30]. Thermogravimetric analysis of Copper (II) Soaps derived from Groundnut (*Arachis hypogaea*) and Sesame (*Sesamum indicum*) edible oils has been studied earlier[31].

Simultaneous fabrication of superhydrophobic cathodic cobalt and anodic Copper surfaces by a one step electrodeposition process has been done earlier[32]. Synthesis, spectral studies, antitubercular and antimicrobial activity of schiff base - Copper (II) complexes have been done[33]. A facile synthesis and biological evaluation of some macrocyclic Copper complexes has been already done[34]. Thermogravimetric analysis of microwave assisted novel macromolecular complexes of metal surfactants has been studied[35].

Synthesis and characterization of Copper complexes with a tridentate nitrogen donor ligand has been done[36]. Studies of solute - solvent interactions and applications of green and blue complexes of Copper (II) palmitate with 2-amino benzothiazoles has been done earlier[37]. Photocatalytic degradation of Copper (II) palmitates in non aqueous media using ZnO as photocatalyst has been studies earlier[38]. Viscometric evaluation of azole derivatives in non - aqueous solvent system has been done earlier[39].

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ENCLOSURE - 3

RESEARCH GAPS IDENTIFIED IN THE PROPOSED FIELD OF INVESTIGATION

Copper Soaps prepared from various edible and non-edible oils have been analysed for their micellar and surface active characteristics. Densities of Copper Soap solutions of various concentrations has been measured and using density data, molar volume and apparent molar volumes has been evaluated[40].

IR, NMR, UV and ESR spectroscopic studies have been also done earlier. The results of the surface tension has been explained on the basis of Szykowski's empirical equation[41]. Thermal stability, kinetic parameters and other fundamental studies will play a significant role in the selection of synthesized Copper Soaps and their complexes in various industries[42].

Photochemistry plays a significant role in many reactions of biological, synthetic and industrial importance in which energy received from sun can be better utilized for converting the pollutants into less toxic materials. Extensive research in photocatalysis resulted in various applications based on the use of semiconductors. The photodegradation of Copper Soaps and their complexes with nitrogen and sulphur containing ligands has various applications. It has been found that plenty of work found need to be done in the proposed field.

Literature survey from different universities and searching through internet concludes that the references on related subject identify the proposed field. Photocatalytic degradation has been considered to be an efficient and rapid process for the degradation of Copper Soaps derived from various edible oils[43]. These Copper Soaps will be thermally analyzed using TGA technique to determine their energy of activation. Biological studies of these compounds will also provide an important account of information about their

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industrial utilization.

The facts mentioned above lead to a curiosity about thermal and photo degradation of Copper Soap complexes derived from edible oils and how to affects by factors like pH, semiconductors, light etc on degradation process. Copper Sesame Soap and Copper Groundnut Soap derived from edible oils and their benzothiazole complexes are being significant due to their toxicity in nature and their biodegradable nature having fungicidal, herbicidal and many other biological activities[53,54].

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ENCLOSURE – 4

MAJOR HYPOTHESIS, IF ANY

Copper Soaps and non-polar solvents have gained a considerable interest and find their uses in various fields of application because of their surface active properties[45-48]. The study of binuclear complexes of these ligands with transition metal Copper (II) is highly interesting due to their significance in physical and bioinorganic chemistry, material science and multi-electron redox chemistry[49].

Due to presence of nitrogen and sulphur atom, the application of benzothiazole and its various derivative as ulcer inhibitor, antiglutamate, fungicidal agents, antibacterial, anti-tubercular agents, herbicidal agents, intermediates in agrochemical and pharmaceutical fields and their uses in diabetic complications have been reported recently by various workers[42].

Literature survey reveals that several methods of kinetic analysis, which have been proposed are based on hypothesis that from single thermogravimetric trace, meaningful values may be obtained for parameters such as activation energy pre-exponential factor and reaction order. Photocatalytic degradation is relatively innovative subject with tremendous potential in the near future because of its environmental applications. The above mentioned facts stimulated our interest to extend the present research work to synthesized complexes and to study their kinetic parameters.

Biological studies of these surfactants will also provide an important account of information about their industrial utilization. The proposed research work will generate new aspects in biological and pharmacological fields due to the biological importance of aforesaid synthesized complexes.

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ENCLOSURE – 5

RESEARCH METHODOLOGY

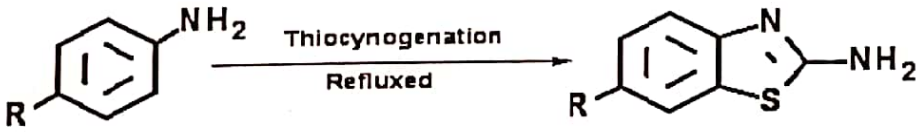
Synthesis

(A) Synthesis of metal soap[50,51,41]

$$RCOOH \xrightarrow[EtOH]{KOH} RCOOK \xrightarrow{Cu^{+2}} (RCOO)_2Cu$$

Copper Soap will be prepared by direct metathesis of the corresponding potassium soap with slight excess of the required amount of copper sulphate solution at 50-55°C. After washing with hot water and alcohol the soap was dried at 100-105°C. Finally under reduced pressure Copper Soap was recrystallize twice from hot benzene.

(B) Synthesis of substituted benzathiazole[52]



The required substituted Benzathiazole will be synthesized THIOCYNATION of aniline in the presence of thiocynation gas, which is generated in situ by the reaction of cupric chloride and ammonium thiocynate. The mixture will be refluxed and stirred for half an hour. After cooling, mixture is neutralized by saturated solution of sodium carbonate. The purity of Benzathiazole has been checked by the thin layer chromatography.

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(C) Synthesis of Complexes

The complexes will be prepared by mixing the metal surfactants and ligand in the molar ratio of 1:1, dissolved in ethanol and benzene and mixture will be reflux for about 1 and half hour. Purity of complex will be checked by TLC.

NMR and ESR spectral studies will be done with help of CDRI Lucknow and IIT Mumbai for structural elucidation.

(D) Thermal and Photocatalytic Degradation and Biological studies

Thermogravimetric methods have been used to study the kinetics of thermal decomposition of soaps and their complexes derived from edible oils. TGA is an analytical technique used to determine a materials thermal stability and its fraction of volatile components by monitoring the weight change that occurs as a specimen is heated.

ZnO and other semiconductors may be used as Photocatalyst to degrade Copper Soaps and their complexes with different ligands. Reaction will be studied spectrophotometrically. The effect of different parameters on the rate of photocatalytic degradation will be observed.

To screen their biological activities and applications of these complexes as antifungal agents, it is proposed to study their toxicity against various fungi.

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ENCLOSURE – 6

LIST OF REFERENCES CITED

1. Sharma R., Heda L.C. and Sharma S., *Tenside Surf. Det.*, **52**(3), 512-516, 2015.

2. Sharma R., and Khan S., *Tenside Surf. Det.*, **46**(3),145-151, 2009.

3. Czeslaw K.K., *Tribology Internet*, **38**, 337, 2005.

4. Zok S., George G., Kalsch W., Nagel R., *Science total environment*, **109-110**, 411, 1991.

5. Maheshwari G. And Singh M., *J. Ind. Chem. Soc.*, **9**, 659-664, 2002.

6. Jagegoud Y. Ijare O.B., Mallikarjuna S.D. and Mruthunjayaswamy B.H.M., *J. Ind. Chem. Soc.*, **79**, 921-924, 2002.

7. Michell A.J., and Holzforschung, **49**(3), 217-221, 1995.

8.Conradie D., Turner P., Conradie W.E., Pendlebury J. And Pizzi T., *Bibliographic Citation Div. Of forest science and technology, Siochholm (Sweden)* 22, 1995.

9. Mehrotra K.N. and Pooranchand, *Tenside Surf. Det.*, **25**(3), 180-185,1988.

10. Mehrotra K.N. and Pooranchand, *Tenside Surf. Det.*, **27**(6), 393-395, 1990.

11. Soyez J.L., *Brighton Crop Protection Conference, "Pests & Diseases"*, Franch, **1**, 451-456, 1992.

12. Sparks C.S. (Cuprinol Ltd Engl.) *Rec. Annu. Conv. Br. Wood Preserv. Assoc.*, 48-51, 1978.

13. Hussain S., Shahid H., and Asjad S., Pakisthan, *J. Sci. Ind. Res.*, **10**(3), 197-200, 1967.

14. Mahapatra B.B. and Mishra R.R., *J. Indian Chem. Soc.*, **78**, 395, 2001.

15. Sharma R., Saxena M., and Sharma N. , *Int. J. Chem. Soc.*, **10**(1), 143-149, 2012.

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16. Mendham J., Denney R.C., Barnes J.D., and Thomas M.J.K. , *Vogel's test book of quantitative chemical analysis*, Addison Wesley Publishing Co., 6th Ed., 2000.

17. Keaitch C., *An Introduction to thermogravimetry*, Heyden, London, 1969.

18. Patton D.L., *Electronic Handout Technique PSC*, 341, L/720, 2008.

19. Sharma R., Heda L.C., Ameta S.C. and Sharma S., *RJPBCS*, **4**(2), 537, 2013.

20. Essien E.A., Appan A.U., Etim U.J., and Udockpo I.U., *Int. R.J. Pure & Applied Chem.*, **6**(2), 95-104, 2015.

21. Myrtle F.J., Zell J.W., *CLIN Chem.* , **21**/10, 1469-1473, 1975.

22. Sharma R., Heda L.C., Bhutra R., and Ojha K.G., *Int. J. Chem. Soc.*, **6**(2), 593-604, 2008.

23. Sharma R., Saxena M., and Acharya S., *j. Ind. Chem. Soc.*, **89**, 585-592, 2012.

24. Sharma R., Tak P., Saxena M., Bhutra R. and Ojha K.G., *Tenside Surf. Det.*, **45**(2), 85-92, 2008.

25. Sharma R., Heda L.C., and Sharma S., *Chem. Sci. & Rev. Lett.*, **4**(13), 7-16, 2015.

26. Goud V.V., Patwardhan A.V., Pradhan N.C., *Int. J. Chem.*, 5(4), 1533-1540, 2007.

27. Ndazi B.S., Nyahumwa C., and Tesha J., *Peer Rev. Article, Bioresources*, **3**(4), 1267-1277.

28. Mathur N., Ahmed I., Kasana A., Bargotya S. And Manna B., *IAAST*, **5**(1), 2014.

29. Mathur N., Rastogi S., and Jain N., *Int. J. Current Reasearch in life sciences*, **3**(12), 70-75, 2014.

30. Owolabi J.B., Alabi K.A., Lajida L., *Sci. Res. Essays*, **10**(23), 649, 2015.

31. Sharma R., Heda L.C., Joram A. and Sharma S., *RJPBCS*, **4**(4), 304, 2013.

Scanned by CamScanner

32*. Jiang X., Hu W., Peng C., Yao K. and Su F., *Materials Research Innovations*, **19**, 83-89, 2015.

33*. Tajudeen S.S. and Kannappan G., *Int. J. Adv. Chem. Sci.*, **4**(1), 40-48, 2016.

34*. Mathur N. and Bargotya S., *IJPSR*, **6**(6), 2538-2545,2015.

35*. Mathur N., Bargotya S. and Mathur R., *J. Appl. Chem.*, **3**(2), 712-719, 2014.

36*. Bussey K.A., Cavalier A.R., Connell J.r., Mraz M.E., Holderread A.S., Oshin K.D. and Pintauer T., *J. Chem. Edu.*, **92**(12), 2140-2145, 2015.

37*. Bargotya S., Manna B., and Kasana A., *IAJPR*, **4**(4), 1981-1988, 2014.

38*. Sharma R., Heda L.C. and Sharma S., *Tenside Surf. Det.*, **52**(6), 2015.

39*. Mathur N., Bargotya S., Saxena P. and Ahmed I., *Chem. Sci. & Rev. Lett.*, **4**(13), 327-334, 2015.

40. Sherwani M.R.K., Sharma R. And Bhutra R., *Int. J. Chem. Sci*,: **6**(2), 735-745, 2008.

41. Mehta V.P., Talesara P.R., Sharma R., Gangwal A. And Bhutra R., *Ind. J. Chem.*, **41A**, 1173-1176, 2002.

42. Tak P., Ph.D. Thesis, *M.D.S. University*, 2008.

43. Sharma S., Ph.D. Thesis, *M.D.S. University*, 2015.

44. Thompson L., *R. Soc. Chem.*, **118**, 56-57, 1992.

45. Li R., Tracy D.J. and Yang J., Dahanayake, Mnilals, *US* 6, 121-222, 2000; 773, 569, 1996.

46. Imori T. And Iida K. Jpn. Kokobi Tokkyo Koho JP 1162, 194, 1999, 97/214, 836, 1997.

47. Stzerndahl M. and Holmberg K., *J. Of Surfactants and Detergents*, **6**(4), 311-318, 2003.

48. Mehrotra K.N. and Jain M., *J. Appl. Polymer Sci.*, **50**(1), 41-47, 1993.

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49. Mathur N., Bargotiya S., and Mathur R., *RJPBCS*, **5**(2), 997, 2014.

50. Mehta V.P., Sharma R. and Ojha K.G., *Tenside Surf. Det.*, **40**, 2003.

51. Sherwani M.R.K., Sharma R., Gangwal A. And Bhutra R., *Ind. J. of Chem.*, Sec A, **42(A)**, (10), 2527-2530, 2003.

52. Adams R., *Organic Reactions*, John Willey & Sons, London, **3**, 1967.

53. O'Brien R.D., Fats and oils, CRC Press, Washington, **2**nd ed., 2000.

54. Filov V.A., Bandman A.L. and Ivin B.A., Harmful *Chemical Substances*, Ellis Horwood Limited, **1**, 1993.

*Recent work references 32-39 added

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ENCLOSURE - 2

REVIEW (WORK ALREADY DONE ON THE SUBJECT OF PROPOSED RESEARCH STUDY)

The research work play an important role in the current scientific development. Plenty of work is going on in various laboratories and industries at national and international level. The physicochemical, analytical, spectral and fungicidal investigations of various edible and non-edible oils have been already done[2,15].

Synthesis, spectroscopic and fungicidal studies of Copper Soap derived from Mustard and Soyabean oils and their urea complexes has been studied earlier[15]. Oil extraction from oilseeds; *Irvingia gabonensis, Citrullus lanatus and Telferia occidentalis* and evaluation in metallic soap preparation have been already done[20]. Simplified photometric Copper-Soap method for rapid assay of serum lipase activity has been investigated[21]. Conductometric studies of Copper (II) Soap in benzene- methanol of various temperature has been already done[22]. Volumetric and viscometric studies of Copper (II) surfactants derived from various edible oils (Sesame, Soyabean, Groundnut, Mustard) in methanol - benzene mixture has been studied earlier[23]. Synthesis and characterisation of antifungal agents containing Copper (II) Soaps and derived from Mustard and Soyabean oil have been done[24].

Degradation kinetics of Copper (II) Soap derived from *Pongamia pinnata* (Karanj) in presence of irradiated semiconducting ZnO has been studied earlier[25]. Kinetic studies on degradation of the oxirane ring of epoxidised Karanj oil have been already done[26].

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